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(54) LUMINESCENT MATERIAL WITH HIGH-SPEED EXCITATION, HIGH LUMINANCE, AND LOW DECAY

(57)Abstract:
PROBLEM TO BE SOLVED: To obtain a luminescent material which has short saturation excitation time and restoration excitation time and a high initial luminance by using a specific baking product.
SOLUTION: This luminescent material with high-speed excitation, high luminance, and low decay is a baking product which is represented by the formula: (SrEu)Al2O4.n[(SrEu)O.(Al1-a-bBbQa)2O3(AlO.OH)] and has an α-alumina content of 50-99% of the total alumina content; a γ-alumina content of 1-50% of the total alumina content; and a Mohs hardness of 6.2-7.5. When (n) is 1, the baking product has a specific gravity of 3.60-3.62; a body color of light yellow green; a luminescent peak wavelength of 518-522 nm: a luminescence color of green; an excitation wavelength of 200-450 nm; an excitation time (103 lux) of 10 min or shorter; an initial luminance of 6,000 mcd/m2 or higher (30 sec after the stopping of excitation); and an afterglow time of 20 hr or longer. In the formula, Q is at least one element selected from among Bi, Ca, Mg, and Mn; $0.0005 \leq a \leq 0.002$: $0.001 \leq b \leq 0.35$; and $1 \leq n \leq 7$.

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CLAIMS

[Claim(s)]
[Claim 1] General formula (SrEu) aluminum2O4 −n [(SrEu) O (aluminum1−a−b B b Q a) and 2O3 (AlO and OH)]
[Q is at least one sort chosen from Bi, calcium, Mg, and Mn among a formula, a is 0.0005<=a<=0.002, b is 0.001<=b<=0.35, and n is 1<=n<=7] −− it is −− the high-speed excitation and quantity brightness low attenuation nature luminescent material characterized by consisting of a baking object.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] Saturation excitation time amount and restoration excitation time amount of this invention are very quick in more detail about high-speed excitation and quantity brightness low attenuation nature luminescent material, and initial brightness is related with very high high-speed excitation and quantity brightness low attenuation nature luminescent material.
[0002]
[Description of the Prior Art] Although the phosphorescence ingredient (ZnS:Cu) obtained by carrying out activation of a sulfide, for example, the zinc sulfide, with copper was known conventionally, from the phosphorescence ingredient of this zinc sulfide system, the ingredient which continues carrying out long duration luminescence whether you are Haruka is developed, and it came to be used for various kinds of applications in recent years. Specifically, invention about high-persistence luminescent material is indicated by China patent application public presentation number CN1053807A. This high-persistence luminescent material is a general formula. m(Sr1-x Eux) O-nAl 2O3 and yB 2O3 It is expressed with [1<=m<=5, 1<=n<=8, 0.001<=y<=0.35]. [however,] After this high-persistence luminescent material uses as a raw material the salts which can generate these oxides after the oxide of the bivalence of aluminum, boron, strontium, and a europium, or heating and calcinates them at 1200 degrees C - 1600 degrees C, it is manufactured according to the process returned according to the reducing atmosphere of nitrogen and hydrogen at 1000 degrees C - 1400 degrees C.
[0003] In addition, as an alumina system baking object which makes boron a constituent, it is indicated by a U.S. Pat. No. 5,376,303 specification, JP,8-170078,A, JP,8-127772,A, etc., and all are the luminescent material which has the high initial brightness and the high-persistence which are excited by source of excitation like sunlight, artificial illumination, an electron ray, or an X-ray.
[0004]
[Problem(s) to be Solved by the Invention] However, although such high-persistence luminescent material had several hours - 10 hours of afterglow time amount in fact, what also has more high initial brightness was fully [practical again] demanded. Then, in order to solve such a trouble, this invention person etc. improved the luminescent material which combined the rare earth metal as the aforementioned aluminate and aforementioned activator of alkaline earth metal of a general formula, and was successful and did patent application to building the luminescent material which brightness becomes from the crystalline which has high-persistence highly further.
[0005] Then, this invention person etc. continues research further about such a luminescent material. When saturation excitation time amount has high brightness and low attenuation nature early extremely also unexpectedly, and declines again as a result of trial-and-error, while luminescent material especially with high brightness is desired, and it is in the condition of low brightness, It succeeded in building the crystalline compound which brightness restores with contact of an instant with excitation ****, and found out that luminescent material with very high brightness was obtained. This invention is made based on this knowledge. Therefore, the

technical problem which this invention tends to solve has very quick excitation, and it is for initial brightness to offer very high high-speed excitation and quantity brightness low attenuation nature luminescent material.
[0008]
[Means for Solving the Problem] Aforementioned The means for solving a technical problem of this invention is the high-speed excitation and a quantity brightness low attenuation nature luminescent material characterized by consisting of a compound expressed with the following general formula.
General formula (SrEu) aluminum2O4 - [(SrEu) O and n(aluminum1-a-b B b Q a)2O 3 (OH)]
[Q is at least one sort chosen from Bi, calcium, Mg, and Mn among a formula, a is 0.0005<=a<=0.002, b is 0.001<=b<=0.35, and n is 1<=n<=7.]
[0007]
[Embodiment of the Invention] Hereafter, when this invention is further explained to a detail, especially luminescent material is an ingredient which continues carrying out long duration luminescence, emitting gradually the energy which excites sunlight, a fluorescent lamp, heat, an impact, etc. by ultraviolet rays, and the crystalline itself has as the light. Initial brightness is high brightness, it is a compound expressed with a general formula, high-speed excitation and quantity brightness low attenuation nature luminescent material of this invention have very quick time amount until excitation will be in a saturation state, and even when it decreases and is in the condition of low brightness, it is a crystalline compound which has the features restored to high brightness by contact of an instant with the source ultraviolet rays of excitation.
[0008] Moreover, high-speed excitation and quantity brightness low attenuation nature luminescent material of this invention A raw material, respectively A strontium compound, the alpha mold aluminum 2O3, and the gamma mold aluminum 2O3 It is the compound chosen from at least one sort of a boron compound, a europium compound and a bismuth compound, a magnesium compound, or a manganese compound. These raw materials are ground. After [raw material] carrying out acid treatment of a kind from the usual acids, such as carbonic acid, at least, Mix all raw materials and the temperature up of this mixture is slowly carried out over 7 - 10 hours under existence of a carbonaceous ingredient from 400 degrees C to 1600 degrees C. It calcinates at 1250 more to 1600 degrees C in 5 hours - 3 hours. Subsequently, after that, It cools and grinds and is characterized by classifying until it becomes 200 degrees C over 7 hours - 10 hours. By this the crystal structure Although it is not clear, it is thought that not a single crystal but the twin crystal crystalline by which triclinic system was piece[of **]-ized is formed in view of what has very quick excitation time amount being obtained. This luminescent material has very quick excitation time amount, and, moreover, it has the luminescent color of from yellowish green until blue.
[0009] By burning temperature's carrying out the temperature up of high-speed excitation and the quantity brightness low attenuation nature luminescent material of this invention slowly over 7 - 10 hours from 400 degrees C to 1250 degrees C, and subsequently calcinating at 1250 more degrees C in 5 hours - 3 hours, it is a baking object in case n of a general formula is 1, and high-speed excitation and quantity brightness low attenuation nature luminescent material of yellowish green - green are obtained for the luminescent color. Moreover, when burning temperature carries out a temperature up slowly over 7 - 10 hours from 400 degrees C to 1600 degrees C and subsequently calcinates at 1600 more degrees C in 5 hours - 3 hours, it is a baking object in case n of a general formula is 2, and high-speed excitation and quantity brightness low attenuation nature luminescent material with the blue luminescent color are obtained.
[0010] The salts to which a strontium compound can generate these oxides with the oxide of the carbonate of strontium and strontium or heating can specifically be used for the manufacture raw material of the high-speed excitation and quantity brightness low attenuation nature luminescent material of this invention. As an oxide of aluminum, what blended alpha mold alumina and the alumina of gamma mold by the specific ratio is used. Moreover, as for a boron compound, the oxide or boric acid of boron is used preferably. Moreover, a europium is used as an activator and the raw material can use the salts which can generate these oxides with the oxide of a

europium, or heating.
[0011] Furthermore, high-speed excitation and quantity brightness low attenuation nature luminescent material of this invention have the **-ized agent, and this **-ized agent absorbs external energy effectively, and is defined as being the matter which transmits this absorbed energy to an emission center. Therefore, a coactivator differs in the operation at the point which is the matter which reinforces the excitation effectiveness in collaboration with an activator. As the aforementioned **-ized agent, Bi, calcium, Mg, and Mn are mentioned and the salts which can generate these oxides with an oxide, a carbonate, or heating can be used as this raw material. As for bismuth compounds (for example, bisumuth oxide etc.) and a lime compound, a calcium carbonate and a calcium oxide are used. A magnesium carbonate and the magnesium oxide of a magnesium compound are desirable, and manganese oxide of a manganese compound is desirable.
[0012] The amount of alpha mold alumina in high-speed excitation and quantity brightness low attenuation nature luminescent material of this invention (alpha-aluminum 2O3) is 50% - 99% of a total amount of an alumina, and the amount of gamma mold alumina (gamma-aluminum 2O3) is 1% - 50% of a total amount of an alumina. In this luminescent material, when it is gamma mold alumina whose percentage of gamma mold alumina and alpha mold alumina is 10 - 20%, what has good, very high brightness and low attenuation nature is obtained.
[0013] the emission peak wavelength of this luminescent material lessens the amount of strontium and a europium -- ** -- what a luminescence peak changes to blue since green, therefore carries out green luminescence when n is 1 is obtained, and when n is 2, what carries out blue luminescence is obtained. Thus, as for the emission peak wavelength of the high brightness high-persistence luminescent material of this invention, it turns out that it changes with number of n, it shifts to a short wavelength side from a long wavelength side as the number becomes large, and it changes from green to blue. Moreover, what has the large number of n is obtained in this case by making burning temperature shift to an elevated temperature little by little.
[0014] The amount of the boron in this invention has the desirable range of 0.001<=b<=0.35, and when b is a small amount from 0.001, it does not have any effect on the manifestation of the low attenuation nature of a crystalline, either. Moreover, since b of a boron oxide system product increases in a baking object when [than 0.35] more and low attenuation nature falls, the amount of boron is not desirable. Moreover, the amounts of the **-ized agent expressed with Q are 0.0005-0.002, this range is desirable, and when the amount of these is a small amount from 0.0005, ****** does not have any effect in the manifestation of the long afterglow phosphorescent ability of a crystalline. Moreover, since the amount included in the hole of a crystalline was decided when there were more amounts of the **-ized agent expressed with Q than 0.002, it is economically useless. High-speed excitation and quantity brightness low attenuation nature luminescent material of this invention are very strong, and the degree of hardness is Mohs hardness 6.2-7.5.
[0015] High-speed excitation and quantity brightness low attenuation nature luminescent material of this invention have the following properties.
[0016] [When n of a general formula is 1]
(1) Specific gravity 3.60-3.62 (2) Color of the body The Asagi green (3) Mohs hardness 6.2-6.5 (4) emission peak wavelength 518-522nm (5) luminescent color Green (6) excitation wavelength 200-450nm(7) excitation time amount (103 lux) (Eight) less than 10-minute initial brightness Two or more (after [an excitation halt] 30 seconds) 6000 mcd/m
(9) Afterglow time amount 20 hours or more [0017] [When n of a general formula is 2]
(1) Specific gravity 3.69-3.71 (2) Color of the body The Asagi green (3) Mohs hardness 7.0-7.5 (4) emission peak wavelength 487-491nm (5) luminescent color Blue (6) excitation wavelength 200-450nm(7) excitation time amount (103 lux) (Eight) less than 30-minute initial brightness Two or more (after [an excitation halt] 30 seconds) 3000 mcd/m
(9) Afterglow time amount 20 hours or more [0018] High-speed excitation and quantity brightness low attenuation nature luminescent material of this invention can manufacture luminescence ink and luminescence resin by mixing in ink or resin. The brightness of the

luminescent material of this invention is very high, and more nearly usable than the conventional luminescent material as an object for a display at night. For example, if it is used for the display of a road, an advertisement, stationery, a toy, sporting goods, etc., the energy which absorbed light and was absorbed by in the dark will be emitted in the form of light, and light will be emitted continuously for 40 to 60 hours or more. It is 20 - 40 hours practical. Moreover, if it is used as a source of a fill-in flash of the back light of liquid crystal, laborsaving of a power source or lightweight-ization of a device can be attained.
[0019] [Operation] High-speed excitation and quantity brightness low attenuation nature luminescent material of this invention have very short excitation time amount by using a **-ized agent together, and the luminescent material of high brightness is obtained in an instant.
[0020]
[Example] Hereafter, although this invention is further explained based on an example, this example is for explaining this invention, and this invention is not limited to this.
[0021] [Example 1] SrCO3 14.48g, alpha-aluminum 2 O3 8.63g, gamma-aluminum 2O3 1.423g, and H3 BO3 0.866g are prepared. BiO(0.0005 mols)0.113g is prepared as a raw material of an activator, respectively as a raw material of Eu2 O30.176(0.0005 mols) g and a **-ized agent. These raw materials were ground in mean particle diameter of 2 micrometers, respectively. Among these, SrCO3 Eu 2O3 After making it 1 in all clue, 10% of aerated water solution is put in, and stirring mixing is carried out. Subsequently, contents are settled from the obtained acid-treatment water solution. After filtering this precipitate and dissociating, this precipitate is dried at 120 degrees C for 4 hours. Thus, the obtained dry matter was ground in mean particle diameter of 2 micrometers. It is alpha-aluminum 2O3 to this desiccation powder. In addition, it mixed enough and uniform mixture was obtained. Furthermore, gamma-aluminum 2O3 which is other above-mentioned raw materials at this mixture and H3 BO3 And BiO is added, and after mixing, it puts into a crucible.
[0022] After introducing this crucible into carbon powder, it puts into an electric furnace, a temperature up is carried out over 8 hours from 400 degrees C to 1250 degrees C, and, subsequently it calcinates at the temperature of 1250 degrees C for 4 hours. Subsequently, after cooling to 200 degrees C over 8 hours, it takes out from an electric furnace. When becoming a room temperature, the ball mill ground, it classified by the screen of 200 more meshes, and the luminescent material (1) of this invention was obtained. the place which was made to excite the obtained luminescent material for 10 minutes 1000 Lux under 27W fluorescent lamp, and measured o'clock of initial brightness for 30 seconds after the excitation halt -- 6000 mcd/m2 it was . 520 micrometers of emission peak wavelengths, and ** -- light was emitted green and the thing of n= 1 of specific gravity 3.60, Mohs hardness 6.2, and the general formula like the following was obtained for afterglow time amount 30 hours.
[0023] (SrEu) aluminum2O4, O, and (aluminum0.9855B0.014 Bi0.0005) 2O 3(AlO and OH)]
[0024] [Example 2] SrCO3 12.397g, CaCO3 1.471g, alpha-aluminum 2O3 8.063g, gamma-aluminum 2 O3 1.423g, and H3 BO3 0.866g are prepared. Mn2 O3 0.047g and BiO0.045g are prepared as a raw material of an Eu2 O3 0.176g and **-ized agent as a raw material of an activator, respectively. These raw materials were ground in mean particle diameter of 2 micrometers, respectively. Among these, SrCO3 Eu 2O3 After making it 1 in all clue, 15% of aerated water solution is put in, and stirring mixing is carried out. Subsequently, contents are settled from the obtained acid-treatment water solution. After filtering this precipitate and dissociating, this precipitate is dried at 80 degrees C - 100 degrees C for 5 hours to 4 hours. Thus, the obtained dry matter was ground in mean particle diameter of 2 micrometers. It is alpha-aluminum 2O3 to this desiccation powder. In addition, it mixed enough and uniform mixture was obtained. Furthermore, gamma-aluminum 2O3 which is other above-mentioned raw materials at this mixture, H3 BO3, and Mn 2O3 And BiO is added, and after mixing, it puts into a crucible.
[0025] After introducing this crucible into carbon powder, it puts into an electric furnace, a temperature up is carried out over 8 hours from 400 degrees C to 1250 degrees C, and, subsequently it calcinates at the temperature of 1250 degrees C for 4 hours. Subsequently, after cooling to 200 degrees C over 8 hours, it takes out from an electric furnace. When becoming a room temperature, the ball mill ground, it classified by the screen of 200 more meshes, and the

luminescent material (2) of this invention was obtained. the obtained luminescent material — 520 micrometers of emission peak wavelengths, and ** — light was emitted green and the thing of n= 1 of specific gravity 3.62, Mohs hardness 6.3, and a general formula was obtained for initial brightness (after [an excitation halt] 30 seconds) 6200 mcd/m2, and excitation time amount (1000 luxs) 8 minutes for afterglow time amount 40 hours.

[0028] (SrEu) aluminum2O4, O, and (aluminum0.9855B0.014 Bi0.0002Mn0.0003) 2O 3(AlO-OH)]

[0027] [Example 3] CaCO3 5.88g, SrCO3 5.78g, alpha-aluminum 2 O3 5.692g, gamma-aluminum 2 O3 3.794g, and H3 BO3 0.866g are prepared. 2O30.039g of Mn and MgCO3 0.021g are prepared as a raw material of an Eu2 O3 0.176g and **-ized agent as a raw material of an activator, respectively. The acid treatment same after grinding the above raw material as an example 1 is performed, and after mixing, it puts into a crucible. After introducing this crucible into carbon powder, it puts into an electric furnace, a temperature up is carried out over 8 hours from 400 degrees C to 1600 degrees C, and, subsequently it calcinates at the temperature of 1600 degrees C for 4 hours. Subsequently, after cooling to 200 degrees C over 8 hours, it takes out from an electric furnace. When becoming a room temperature, the ball mill ground, it classified by the screen of 200 more meshes, and the luminescent material (3) of this invention was obtained. As for the obtained luminescent material, the outstanding blue thing beyond afterglow time amount 50 hour (thing of n= 2) was obtained for 490nm of emission peak wavelengths, initial brightness 3200 mcd/m2, and excitation time amount (1000 luxs) 30 minutes.

[0028] (SrEu) aluminum 2O4-(SrEu) O-2 [(aluminum0.9855B0.014 Mn0.0003Mg0.0002) 2O3 (AlO and OH)]

[0029] [Example 4] CaCO3 8.337g, SrCO3 2.070g, alpha-aluminum 2 O3 8.063g, gamma-aluminum 2 O3 1.423g, and H3 BO3 0.866g are prepared. As a raw material of an activator, Mn2 O3 0.03g and BiO0.113g are prepared as a raw material of an Eu2 O3 0.176g and **-ized agent, respectively. The acid treatment same after grinding the above raw material as an example 1 is performed, and after mixing, it puts into a crucible. The crucible into which this mixture was put was manufactured like the approach of an example 3, and the luminescent material (4) of this invention was obtained. As for the obtained luminescent material, the outstanding blue thing beyond afterglow time amount 50 hour (thing of n= 2) was obtained for 490nm of emission peak wavelengths, initial brightness 3200 mcd/m2, and excitation time amount (1000 luxs) 30 minutes.

[0030] (SrEu) aluminum 2O4-(SrEu) O-2 [(aluminum0.9853B0.014 Bi0.0003Mn0.0002) 2O3 (AlO and OH)]

[0031] [Example 5] SrCO3 2.170g, CaCO3 8.337g, alpha-aluminum 2 O3 16.126g, gamma-aluminum 2 O3 2.846g, and H3 BO3 1.732g are prepared. As a raw material of an activator, BiO0.045g and MgCO3 0.025g are prepared as a raw material of an Eu2 O3 0.176g and **-ized agent, respectively. The acid treatment same after grinding the above raw material as an example 1 is performed, and after mixing, it puts into a crucible.

[0032] The temperature up of this crucible is carried out over 10 hours from 400 degrees C to 1600 degrees C, and it calcinates at the temperature of 1600 more degrees C for 4 hours, and after that, it cools to 200 degrees C over 8 hours, and, subsequently takes out from an electric furnace. The ball mill ground at the room temperature, it classified by the screen of 200 meshes, and the luminescent material (5) of this invention was obtained. This luminescent material was excited for 30 minutes 800 Lux under 27W fluorescent lamp, the initial brightness from o'clock was measured for 5 seconds after the excitation halt, and it was shown in Table 1. Moreover, this luminescent material emitted light blue 490nm of emission peak wavelengths, and specific gravity 3.7, Mohs hardness 7.3, and the thing of n= 2 were obtained for afterglow time amount 30 hours.

[0033] (SrEu) aluminum 2O4-(SrEu) O-2 [(aluminum0.9855B0.014 Bi0.0002Mg0.0003) 2O3 (AlO and OH)]

[0034]

table 1 [ ] Initial brightness Excitation time amount (800Lux) 5 seconds 10 seconds 30 seconds 1 Part 5 Part 10 minutes 20 minutes Example 2 of a 30-minute comparison After an excitation halt 110 160 480 920 2100 2260 2480 2440 (mcd/m2) Saturation achievement quotient 4.5 6.6 19.7 37.7 86.1 92.6 100 (%) example 5 After an excitation halt 640 900 2210 2630 4120 4630 4610

(mcd/m2) Saturation achievement quotient 13.919.5 47.9 57.0 89.4 100 (%) [0035] [Example 1 of a comparison] SrCO3 14.73g, alpha-aluminum 2 O3 9.93g, gamma-aluminum 2O3 0.10g, and H3 BO3 0.22g are prepared. As a raw material of an activator and an addition activator, Eu2 O3 0.035g and Dy2 O3 0.037g are prepared, respectively. The above raw material is ground, respectively, and after mixing, it puts into a crucible. After introducing the crucible into which the above-mentioned mixture was put into carbon powder, it puts into an electric furnace, a temperature up is carried out over 8 hours from 400 degrees C to 1250 degrees C, and, subsequently it calcinates at the temperature of 1250 degrees C for 4 hours. Subsequently, after cooling to 200 degrees C over 8 hours, it takes out from an electric furnace. When becoming a room temperature, the ball mill ground, it classified by the screen of 200 more meshes, and the luminescent material for a comparison (6) was obtained. this luminescent material — 520 micrometers of emission peak wavelengths, and ** — light was emitted green and specific gravity 3.60, Mohs hardness 6.2, and the thing of n= 1 were obtained for initial brightness 4500 mcd/m2, and excitation time amount (1000 luxs) 30 minutes for afterglow time amount 40 hours.

[0036] [Example 2 of a comparison] SrCO3 14.73g, alpha-aluminum 2 O3 13.71g, gamma-aluminum 2 O3 5.86g, and H3 BO3 0.94g are prepared. As a raw material of an activator and an addition activator, Eu2 O3 0.035g and Dy2 O3 0.075g are prepared, respectively. The above raw material is ground, and after mixing, it puts into a crucible. The temperature up of the crucible into which this mixture was put is carried out over 10 hours from 400 degrees C to 1600 degrees C, and it calcinates at the temperature of 1600 more degrees C for 4 hours, and after that, it cools to 200 degrees C over 8 hours, and, subsequently takes out from an electric furnace. The ball mill ground at the room temperature, it classified by the screen of 200 meshes, and the luminescent material for a comparison (7) was obtained. This luminescent material was excited for 30 minutes 800 Lux under 27W fluorescent lamp, and the initial brightness from o'clock was measured for 5 seconds after the excitation halt. Moreover, this luminescent material emitted light blue 489 micrometers of emission peak wavelengths, and Mohs hardness 7.3 and the thing of n= 2 were obtained for afterglow time amount 50 hours. The obtained result is shown in Table 1.

[0037]

[Effect of the Invention] With the compound expressed with a general formula, initial brightness is high, excitation is very quick, and it excels in low attenuation nature, and high-speed excitation and quantity brightness low attenuation nature luminescent material of this invention emit light yellowish green thru/or blue.

[Translation done.]